

### Remarks

Further and favorable reconsideration is respectfully requested in view of the foregoing amendments and following remarks.

Thus, referring to the first paragraph on page 4 of the Office Action, each of claims 1 and 2 has been amended to indicate that the composition of the carburizing gas is adjusted such that the (H/C) ratio of hydrogen atoms (H) to carbon atoms (C) is  $1 \leq H/C \leq 9$ . Similar language is used in the sentence bridging pages 3-4, page 8, line 9 and Table 4 on page 18 of the specification.

The patentability of the presently claimed invention over the disclosure of the reference relied upon by the Examiner in rejecting the claims will be apparent upon consideration of the following remarks.

Thus, the rejection of claims 1 and 2 under 35 U.S.C. §103(a) as being unpatentable over Sato et al. is respectfully traversed.

According to the presently claimed invention, because plasma carburizing is carried out in an atmosphere of 400 to 690°C comprising a carburizing gas having a composition thereof adjusted such that an (H/C) ratio of hydrogen atoms (H) to carbon atoms (C) is  $1 \leq H/C \leq 9$ , an  $\alpha$ -layer deposits on the surface of the titanium metal in which  $\alpha$ -type (hexagonal system) and  $\beta$ -type (regular system) structures coexist, so that it is possible to turn many carbon atoms to solid solution on the surface to the limit of the  $\alpha$ -type titanium metal at the predetermined temperature of plasma carburizing without the need for aging before carburizing. (Please see the paragraph bridging pages 4-5 of the specification.)

That is, according to the inventions of claims 1 and 2, it is possible to form a relatively thick and durable sliding treatment layer on the surface of a titanium metal and thus to reduce the friction coefficient and the amount of wear in a stable manner, without the possibility of deposition of soot and glasslike carbon on the surface of the titanium metal. (Please see page 13, line 13 to page 14, line 4 of the specification.)

Sato et al merely teach plasma carburizing carried out in an atmosphere of 0.5 to 15 Torr and 700 to 1100°C, and is silent about the feature of the present invention wherein plasma carburizing is carried out in an atmosphere of 400 to 690°C comprising a

carburizing gas having a composition thereof adjusted such that the (H/C) ratio of hydrogen atoms (H) to carbon atoms (C) is  $1 \leq H/C \leq 9$ .

In Examples 1-2 of Sato et al. carburizing is carried out in an atmosphere comprising 100% propane gas. The (H/C) ratio of 100% propane gas is 0.2 and thus is out of the range of the claimed (H/C) ratio.

The (H/C) ratio of 100% is the parameter frequently used in the field of analysis of organic elements. This value is obtained in a simple manner by use of a well-known element analyzer by separating nitrogen, water and carbon dioxide from organic combustion gas to be measured, absorbing them, and detecting them in respective thermal conductivity detectors to analyze elements.

The (H/C) ratio is sometimes also referred to as "molar ratio". But the molar ratio or the (H/C) ratio as used in the present invention is not the mere ratio between the numbers of atoms of two elements. This is apparent from Comparative Example 4 of Table 4 on page 18 of the specification, in which the gas flow rate of  $C_3H_8$  is 300 and the gas flow rate of  $H_2$  is zero, which means that the atmospheric gas comprises 100% propane gas (as in Sato et al.), and the H/C ratio of the 100% propane gas is shown as 0.22, not  $8/3$  or 2.67 as indicated by the Examiner.

More specifically, the (H/C) ratio of 100% propane gas ( $C_3H_8$ ) is calculated by the following equation:

$$(1 \text{ (atomic weight of hydrogen)} \times 8) / (12 \text{ (atomic weight of carbon)} \times 3) = 8/36 = 0.222....$$

Thus, the (H/C) ratio of the atmospheric gas of Sato et al., which comprises 100% propane gas, is far below the claimed range of the (H/C) ratio. It is therefore apparent that it is impossible to turn many carbon atoms to solid solution on the surface to the limit of the  $\alpha$ -type titanium metal to form a relatively thick, durable sliding treated surface on the surface of the titanium metal without the possibility of deposition of soot or glass-like carbon on the surface of the titanium metal.

For these reasons, Applicants take the position that the presently claimed invention is clearly patentable over the applied reference.

Therefore, in view of the foregoing amendments and remarks, it is submitted that the ground of rejection set forth by the Examiner has been overcome, and that the application is in condition for allowance. Such allowance is solicited.


Telephone Discussion with Examiner

The last paragraph on page 5 of the Amendment filed July 24, 2006 refers to "a reference". During a telephone discussion with the Examiner on January 8, 2007, the Examiner confirmed that no attachment to the Amendment was scanned into the PTO Image File Wrapper. Accordingly, a copy of the reference, referred to as a Military Specification for "HEAT TREATMENT OF TITANIUM AND TITANIUM ALLOYS", is submitted herewith.

Respectfully submitted,

Eiichi ISHII et al.

By:

  
Michael R. Davis  
Registration No. 25,134  
Attorney for Applicants

MRD/pth  
Washington, D.C. 20006-1021  
Telephone (202) 721-8200  
Facsimile (202) 721-8250  
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NOT MEASUREMENT SENSITIVE

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13 January 1991  
SUPERSEDING  
MIL-H-81200A  
12 September 1968

## MILITARY SPECIFICATION

### HEAT TREATMENT OF TITANIUM AND TITANIUM ALLOYS

This specification has been approved for use by all Departments and Agencies of the Department of Defense.

#### 1. SCOPE

1.1 Purpose. This specification covers the heat treatment of titanium and titanium alloy products, including wrought and cast products, by material producers. This specification also covers furnace equipment requirements, test procedures, and general information for heat treating procedures, heat treating temperatures, and material test procedures for the heat treatment of titanium and titanium alloys. It also describes procedures which, when followed, have produced the desired properties within the limitations of the respective alloys.

1.2 Heat treatments. The heat treatments covered by this specification are:

Anneal	Solution heat treatment
Beta anneal	Beta solution heat treatment
Recrystallization anneal	Age
Duplex anneal	Stress relief

1.3 Alloys. In addition to Commercially Pure Titanium (Ti40, Ti55, and Ti70), the following titanium alloys are covered by this specification:

<u>Alpha alloys</u>	<u>Alpha-Beta alloys</u>	<u>Beta alloys</u>
6Al-2Sn-4Zr-2Mo	6Al-4V	13V-11Cr-3Al
5Al-2.5Sn	6Al-4V ELI	3Al-8V-6Cr-4Mo-4Zr
5Al-2.5Sn ELI	6Al-6V-2Sn	15V-3Al-3Cr-3Sn
6Al-2Cb-1Ta-0.8Mo	3Al-2.5V	10V-2Fe-3Al
8Al-1Mo-1V	6Al-2Sn-4Zr-6Mo	
11Sn-5Zr-2Al-1Mo	6Al-2Sn-2Zr-2Mo-2Cr-0.25Si	
	5Al-2Sn-2Zr-4Mo-4Cr	

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Systems Engineering and Standardization Department (Code 53), Naval Air Engineering Center, Lakehurst, NJ 08733-5100 by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

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FSC 95GP

## 2. APPLICABLE DOCUMENTS

### 2.1 Government documents.

2.1.1 Standards and handbooks. The following standard forms a part of this document to the extent specified herein. Unless otherwise specified, the issue of this document shall be that listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation (see 6.2).

#### STANDARDS

##### MILITARY

MIL-STD-45662 Calibration System Requirement

(Unless otherwise indicated, copies of federal and military specifications, standards, and handbooks are available from the Standardization Documents Order Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.)

2.2 Non-Government publications. The following document(s) form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of the documents not listed in the DODISS are the issues of the documents cited in the solicitation.

#### AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM E 3	Metallographic Specimens, Preparation of.
ASTM E 8	Tension Testing of Metallic Materials.
ASTM E 146	Chemical Analysis of Zirconium and Zirconium Alloys.
ASTM E 290	Semi-Guided Bend Test for Ductility of Metallic Materials.

(Applications for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103-1187.)

#### SOCIETY OF AUTOMOTIVE ENGINEERS, INC. (SAE) (Aerospace Materials Specifications)

AMS-2750	Pyrometry
AMS-2801	Heat Treatment of Titanium Alloy Parts

(Applications for copies should be addressed to SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.)

(Non-government standards and other publications are normally available from the organizations which prepare or distribute the documents. These documents may also be available in or through libraries or other informational services.)

2.3 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document shall

take precedence. Nothing in this document, however, shall supersede applicable laws and regulations, unless a specific exemption has been obtained.

### 3. REQUIREMENTS

**3.1 General.** All heating and quenching equipment and procedures applied shall yield products complying with the requirements of appropriate acquisition documents. Equipment and procedures shall be designed to minimize the introduction of hydrogen, oxygen, nitrogen or other contaminants and in any case shall not allow introduction beyond levels established by the acquisition documents. Deviation from process requirements specified herein or the application of processes different from those contained herein, may be used provided that compliant products result, these exceptions have been proven satisfactory, and that they are made known to the purchaser with accompanying data or other justification to support the deviation prior to application of the deviant process.

**3.1.1 Heat treatment of mill products and titanium alloy parts.** The requirements specified herein are applicable to the heat treatment of mill products (see 6.4.1). Parts (see 6.4.2) may be heat treated in accordance with the requirements of AMS-2801. When parts are heat treated as specified herein, bend properties (see 3.7.2) and bend tests (see 4.7.2) shall not apply. The rework of parts, when applicable, shall be in accordance with AMS-2801.

### 3.2 Heating systems.

#### 3.2.1 Batch furnaces.

**3.2.1.1 General requirements.** Such furnaces may employ electrical heating elements or fuel combustion as heat sources. Muffle furnaces and retorts are also allowed. Allowable environments surrounding the furnace charge during heating are: inert gas (argon or helium), vacuum, slightly oxidizing mixtures resulting from the combustion in air of hydrocarbons (gas or oil), and air itself. When removal of surface contamination is not feasible, inert gas or vacuum environment shall be employed. The selection of an atmosphere shall be such as to establish conformance with 3.1.

**3.2.1.2 Inert gases.** Inert gases within the furnace shall be circulated as necessary to protect all surfaces of the workpieces comprising the furnace charge. The dew point of the inert gases shall be minus 65°F (-54°C) or lower. This requirement shall be met during all stages of a heating, soaking or cooling cycle. Ducts and zones which are to contain furnace charges shall be so sealed as to prevent contamination of any charge to the degree that it is rendered nonconforming to specified material requirements.

**3.2.1.3 Vacuum.** Vacuum furnaces used for outgassing hydrogen shall be capable of reducing hydrogen concentrations within the charge to levels complying with 3.1. Vacuum furnaces and retorts used for prevention of surface contamination shall be capable of yielding product conforming to 3.1.

**3.2.1.4 Combusted hydrocarbons.** Furnaces heated by the combustion in air of gas or oil shall contain a slightly oxidizing gas mixture. There shall be no impingement of flame upon the furnace charge.

3.2.1.5 Prohibited atmospheres. Endothermic, exothermic, hydrogen, and cracked ammonia atmospheres shall not be used during any heat treatment operation.

3.2.1.6 Furnace purging. Prior to thermal treatment of workpieces, each furnace which has contained an atmosphere unacceptable for heat treating (see 3.2.1.5) shall be purged with air or inert gas, as applicable.

3.2.1.6.1 Purging prior to introducing air or combusted hydrocarbons. The volume of purging air introduced shall be at least twice the volume of the furnace chamber. During purging, the minimum temperature within the chamber shall be the intended soaking temperature of the charge. When air-flow purging is impractical, the furnace temperature shall be set at 200°F above the intended soaking temperature, and be held at that temperature for a minimum of four hours. Following purging, the furnace shall be stabilized at the required temperature, charged, and the charge heated and soaked in accordance with 3.2.1.7, as applicable. Following the thermal treatment and any subsequent cleaning, pickling, or other process which may introduce hydrogen contamination, specimens shall be taken from the charge and subjected to the test specified in 4.7.3. Results shall show compliance with 3.6.5, as applicable.

3.2.1.6.2 Purging prior to introducing inert gas. Procedures for purging shall comply with 3.2.1.2. The volume of gas introduced shall be at least twice the volume of the furnace chamber. Furnaces shall be charged while cold, and then purged and filled with inert gas. The charge shall then be heated and soaked in accordance with 3.2.1.7, as applicable. Following the thermal treatment, samples shall be taken from the charge and subjected to the test specified in 4.7.3. Results shall show conformance to 3.6.5, as applicable. Additionally, samples shall be taken from the charge and examined in accordance with 4.7.4.2. Results shall show conformance to 3.6.6.

3.2.1.7 Temperature uniformity. Batch furnaces shall be so controlled that, during heating and soaking periods, temperatures at all points within the working zones are less than the maxima of the ranges specified in Tables I, III, IV, and V, as applicable to the product. After a charge has reached a pre-selected soaking temperature throughout its thickness within a specified range, the temperature at any point in the working zone shall lie within the limits specified below, as applicable to the thermal treatment intended. Regardless of temperature tolerances, no soaking temperature during any thermal treatment shall be higher than the applicable maximum, nor lower than the applicable minimum of the specified range.

<u>Heat treatment</u>	<u>Temperature tolerance</u>	
	<u>°F</u>	<u>°C</u>
Annealing	±25	±14
Beta annealing or beta solution heat treating	±25	±14
Recrystallization annealing	±25	±14
Duplex annealing	±25	±14
Solution heat treating	±25	±14
Stress relieving	±25	±14
Aging	±15	±8

### 3.2.2 Continuous furnaces.

3.2.2.1 General requirements. Such furnaces may be heated by radiation from electrically-energized heating elements or by combusted hydrocarbons.

3.2.2.2 Temperature control. A temperature profile from furnace entry to exit shall be so developed and maintained that the charge within the working zone experiences the appropriate thermal cycle to the degree necessary for eventual product acceptability in terms of specified requirements.

3.2.2.3 Continuous vacuum furnaces. Continuous vacuum furnaces shall be so sealed as to minimize hydrogen, oxygen and nitrogen absorption of the product and in any case shall not allow absorption beyond levels established by the acquisition documents.

3.2.3 Continuous induction heating. Such a heating method shall be applied only to the annealing of thin-walled tubing and extrusions of thin sections. The technique shall be such that the workpiece being heated is of uniform temperature around the perimeter of its cross-section. Prior to production, values of the process parameters which produce acceptable product shall be determined and documented.

3.2.4 Pyrometry and furnace temperatures control. The requirements and procedures for control and testing of furnaces, ovens, vacuum furnaces and allied pyrometric equipment used for heat treatment shall be in accordance with AMS-2750.

3.2.4.1 System accuracy. Each system shall be set to control working temperatures and be corrected to within the applicable tolerances specified herein.

### 3.3 Quenching facilities and media.

3.3.1 Quenching baths. Quenching baths holding water or oil shall be of such dimensions, volume, and construction that products quenched therein will, upon aging, develop the properties specified within applicable product documents. Mechanical stirring of the bath may be applied when necessary.

3.3.2 Spray or flow quenching. Continuous furnaces discharging solution heat treated alloy sheet, plate, and strip may be equipped with a quenchant system which directs a spray or streams of quenchant onto the product as it emerges from the furnace. The spray or flow of quenchant shall be applied evenly over the workpiece width, top and bottom surfaces, over a period of time and at a volume rate such that the resulting product will upon aging develop properties meeting specified requirements.

3.3.3 Location of quenching facility. Quenching and handling facilities shall be located such that contact between quenchant and workpieces occurs within the time required for compliance with 3.1 and for the Ti-6Al-4V and Ti-6Al-4V ELI alloys, within the limits specified in Table II.

3.3.4 Quenching media. Use of molten salt baths for quenching is prohibited.



3.4 Ancillary equipment. Jigs, fixtures, trays, hangers, racks, ventilators, etc. shall be so designed and constructed that each workpiece can be processed in accordance with this specification.

3.5 Thermal treatment parameter values. The parameters (ie. temperatures, times, etc.) for the various thermal treatment processes shall be as specified herein, except where deviation has been accepted by the purchaser in accordance with 3.1 (see 6.2).

3.5.1 Solution heat treating. Solution heat treating of parts, mill product, castings, and forgings shall be as specified in Table I, as applicable.

3.5.2 Quenching. All heat treatable titanium alloys, except alloys which can be cooled in air or inert gas, shall be quenched by complete immersion in water or oil, as applicable, or by water spray or flow when applicable to quenching sheet, strip or plate. Maximum delay times for Ti-6Al-4V and Ti-6Al-4V ELI alloys shall conform to Table II, and for other alloys shall be as necessary to develop required properties.

3.5.3 Aging. Solution heat treated alloy workpieces shall be aged in accordance with Table III, as applicable. Workpieces shall be cooled from the aging temperature in air, an inert gas, or in the aging furnace.

3.5.4 Stress-relieving treatment. Time-temperature cycles for stress relieving shall be as specified in Table IV, as applicable. Workpieces may be cooled from the stress relieving temperature in air, an inert gas, or in the stress-relieving furnace.

3.5.5 Annealing. Time-temperature cycles for annealing shall be as specified in Table V, as applicable. For duplex annealing of Ti-6Al-4V and Ti-6Al-4V ELI alloys, see Table V, note 6/.

3.5.6 Beta annealing. When such annealing or beta solution heat treatment is specified (see 6.2), a lot of workpieces of Ti-6Al-4V, Ti-6Al-4V ELI, Ti-6Al-6V-2Sn, or other alpha-beta alloy shall be soaked at a temperature which is  $50 \pm 25^\circ\text{F}$  ( $30 \pm 15^\circ\text{C}$ ) above the determined beta transus of the lot (see 4.7.4.1). The soaking time shall be such that all portions of the furnace charge and of each workpiece including midsection are soaked for at least 30 minutes. Following soaking, the lot shall be cooled in air or inert gas to ambient temperature. Furnace cooling is not permitted. Water quenching shall not be performed, unless specified in the contract or on the drawing. When water quenching is specified, the products of Ti-6Al-4V, Ti-6Al-4V ELI, and Ti-6Al-6V-2Sn shall be given a second anneal between  $1350^\circ\text{F}$  ( $732^\circ\text{C}$ ) and  $1400^\circ\text{F}$  ( $760^\circ\text{C}$ ) for 1 to 3 hours.

3.6 Process requirements other than those specified in 3.5.

3.6.1 General requirements. All heating, quenching and other processing equipment used for thermal treating shall be capable of producing end product conforming to 3.1. All units of a lot shall be heated uniformly and on the

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TABLE III. Aging schedule.

Material	Aging temperature °F °C		Soaking times, hours 1/	
Alpha Alloys				
8Al-1Mo-1V 11Sn-5Zr-2Al-1Mo	1000-1150 900-1000	540-620 480-540	8 - 24 20 - 30	2/ 2/ 3/
Alpha-Beta Alloys				
6Al-4V 6Al-6V-2Sn 6Al-2Sn-4Zr-2Mo 6Al-2Sn-4Zr-6Mo 6Al-2Sn-2Zr-2Mo-2Cr-0.25Si 5Al-2Sn-2Zr-4Mo-4Cr	900-1275 <sup>a</sup> 875-1150 1050-1150 1050-1250 900-1250 1100-1250	480-690 470-620 565-620 480-675 480-675 480-675	2 - 8 2 - 10 2 - 8 4 - 8 2 - 10 4 - 8	2/ 3/  2/   3/
Beta Alloys				
13V-11Cr-3Al 4/ 3Al-8V-6Cr-4Mo-4Zr 15V-3Al-3Cr-3Sn 10V-2Fe-3Al	825-975 875-1150 900-1250 900-1150	440-525 470-620 480-675 480-620	2 - 60 2 - 24 2 - 24 8 - 10	3/ 3/ 3/ 3/

Notes:

- 1/ See note 1/, Table I, for definition of soaking time and paragraph 6.3.7.
- 2/ See Table V for duplex annealing. An 8-hour stabilizing treatment at 1050-1100°F (565-595°C) can be considered an aging treatment.
- 3/ Aging time and temperature depends on strength level desired.
- 4/ Springs may be aged at 800°F (425°C).